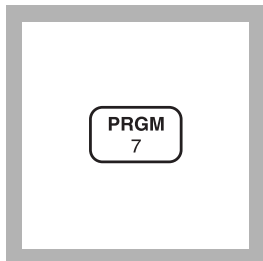


# MOLYBDENUM, MOLYBDATE, Low Range (0 to 3.00 mg/L)

## Ternary Complex Method

For boiler and cooling tower waters



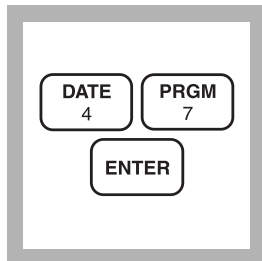
1. Enter the stored program number for molybdate molybdenum.

Press: **PRGM**

The display will show:

**PRGM ?**

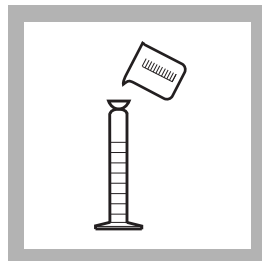
*Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).*



2. Press: **47 ENTER**

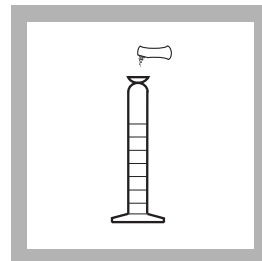
The display will show **mg/L, Mo6** and the **ZERO** icon.

*Note: For alternate forms (MoO<sub>4</sub>), press the **CONC** key.*

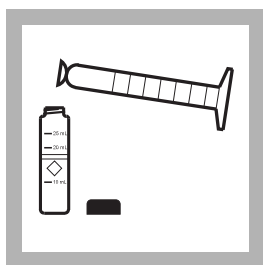


3. Fill a 25-mL mixing graduated cylinder with 20 mL of the sample.

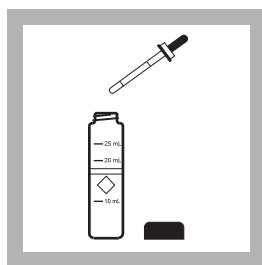
*Note: Filter turbid samples using the labware listed under Optional Apparatus.*



4. Add the contents of one Molybdenum 1 Reagent Powder Pillow to the graduated cylinder. Stopper. Invert the graduated cylinder several times to dissolve the reagents.

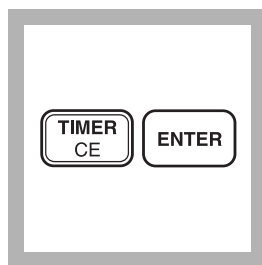


5. Pour 10 mL of the solution into a sample cell.



6. Add 0.5 mL of Molybdenum 2 Reagent to the sample cell. Swirl to mix. This is the prepared sample.

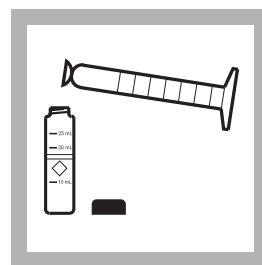
*Note: Molybdenum will cause a green color to form.*



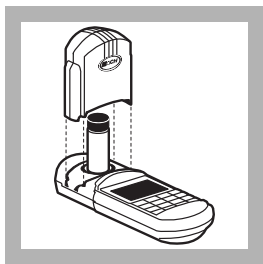
7. Press:

**TIMER ENTER**

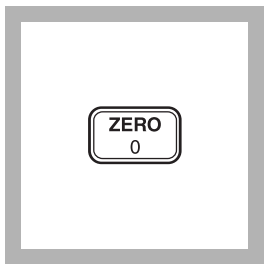
A two-minute reaction period will begin.



8. Fill a second sample cell with 10 mL of solution from the graduated cylinder (the blank).



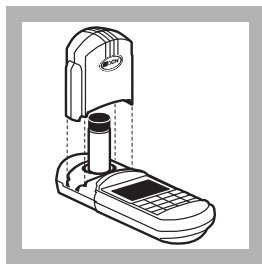
9. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



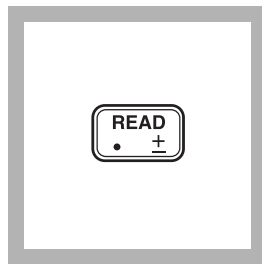
10. Press: **ZERO**  
The cursor will move to the right, then the display will show:

**0.00 mg/L Mo<sub>6</sub>**

*Note: If Reagent Blank Correction is on, the display may flash "limit" (see Section 1).*



11. Place the developed sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**  
The cursor will move to the right, then the result in mg/L molybdate molybdenum will be displayed.

*Note: Standard Adjust may be performed using a prepared standard (see Section 1).*

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## Sampling and Storage

Collect samples in glass or plastic bottles.

## Accuracy Check

### Standard Addition Method

- Add 25 mL of sample to three 25-mL mixing cylinders.
- Snap the neck off a Molybdenum PourRite Ampule Standard Solution, 75 mg/L Mo<sup>6+</sup>.
- Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to three 25-mL samples. Mix thoroughly.
- Analyze 20 mL of each spiked sample as described in the procedure. The molybdenum concentration reading should increase by 0.3 mg/L for each 0.1 mL addition of standard.
- If these increases do not occur, see *Standard Additions* in Section 1 for more information.

### Standard Solution Method

Prepare a 2.0-mg/L molybdenum standard solution by pipetting 10 mL of a 10-mg/L Molybdenum Standard Solution into a 50-

mL graduated mixing cylinder. Dilute to the mark with deionized water and mix thoroughly. Analyze 20 mL of this solution according to the procedure.

## Method Performance

### Precision

In a single laboratory using standard solutions of 2.00 mg/L Mo<sup>6+</sup> and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ±0.009 mg/L Mo<sup>6+</sup>.

### Estimated Detection Limit

The estimated detection limit for program 47 is 0.07 mg/L Mo<sup>6+</sup>. For more information on the estimated detection limit, see *Section 1*.

## Interferences

Interference studies were conducted by preparing a molybdenum standard solution (2 mg/L Mo<sup>6+</sup>) as well as a solution of the potential interfering ion. When the standard solution concentration changed by ±5% with a given ion concentration, the ion was considered an interference.

**Table 1 Negative Interferences**

Ion	Level above which it interferes (mg/L)
Iron	200
Copper	98
Chromium (Cr <sup>6+</sup> )	4.5 <sup>1</sup>
Chloride	1,400
AMP (Phosphonate)	15
Phosphonohydroxyacetic Acid	32
Bisulfate	3,300
Nitrite	350
Aluminum	2
Acrylates	790
Alum	7
Lignin Sulfonate	105
Orthophosphate	4,500
Bicarbonate	5,650
EDTA	1,500
Borate	5,250
Ethylene Glycol	2% (by volume)
Sulfite	6,500
Diethanoldithiocarbamate	32

**Table 1 Negative Interferences (continued)**

Ion	Level above which it interferes (mg/L)
<b>Positive Interferences</b>	
Carbonate	1,325
Silica	600
Benzotriazole	210
Morpholine	6

<sup>1</sup> Read molybdenum concentration immediately after the completion of the two-minute reaction period.

**Table 2 No Interference**

Ion	Highest Concentration Tested (mg/L)
Zinc	400
Calcium	720
Magnesium	8,000
Manganese	1,600
Chlorine	7.5
PBTC (phosphonate)	500
Sulfate	12,800
Bisulfite	9,600
Nickel	250

Phosphonate HEDP at concentrations up to 30 mg/L will increase the apparent molybdenum concentration reading by approximately 10% (positive interference). For these samples, multiply the value obtained in step 12 by 0.9 to obtain the actual molybdenum concentration. As the concentration of HEDP increases above 30 mg/L, a decrease in the molybdenum concentration reading occurs (negative interference).

Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagent and require pretreatment. Adjust the sample pH to 3-5 (use a pH meter or pH paper) by adding drops of an acid or base such as 1.0 N Sulfuric Acid Standard Solution, or 1.0 N Sodium Hydroxide Standard Solution. If a significant volume of acid or base is used, correct the result by dividing the total volume (sample + acid + base) by the original volume and multiplying the test result by this factor.

# MOLYBDENUM, MOLYBDATE, LR, continued

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Large interferences are caused by some biocides used in cooling tower samples. Hach recommends testing the ternary complex procedure on molybdenum standards containing the specific biocides in use to determine if the ternary complex method will work with these samples.

After many samples have been analyzed, the sample cells may show a slight blue color. Rinse with Hydrochloric Acid Solution, 1:1, to eliminate the build-up.

## Summary of Method

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and sensitizing agent to give a stable blue complex.

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## REQUIRED REAGENTS

Molybdenum Reagent Set, 20 mL sample (100 tests) .....24494-00  
Includes: (1) 23524-49, (1) 23525-12

Description	Quantity Required		
	Per Test	Unit	Cat. No.
Molybdenum 1 Reagent for 20 mL sample size .....	1 pillow .....	100/pkg .....	23524-49
Molybdenum 2 Reagent Solution.....	0.5 mL ....	.50 mL MDB .....	23525-12

## REQUIRED APPARATUS

Cylinder, mixing, graduated, 25 mL ..... 1 .....each ..... 1896-40  
Sample Cell, 10-20-25 mL, w/cap ..... 2 .....6/pkg .....24019-06

## OPTIONAL REAGENTS

Hydrochloric Acid Solution, 1:1, 6.0 N ..... 500 mL .....884-49  
Molybdenum Standard Solution, Ampule  
75 mg/L Mo<sup>6+</sup>, 2 mL .....20/pkg .....25575-20  
Molybdenum Standard Solution, 10 mg/L Mo<sup>6+</sup> ..... 100 mL .....14187-42  
Sodium Hydroxide Standard Solution, 1.0 N..... 100 mL MDB .....1045-32  
Water, deionized .....4 L .....272-56

# MOLYBDENUM, MOLYBDATE, LR, continued

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## OPTIONAL APPARATUS

Description	Quantity Required	Unit	Cat. No.
	Per Test		
Cylinder, mixing, graduated, 50 mL .....		each.....	1896-41
Filter Paper, folded, 12.5 cm.....		100/pkg.....	1894-57
Funnel, poly, 65 mm .....		each.....	1083-67
pH Paper, 1-11 pH units.....		5 rolls/pkg.....	391-33
Pipet, TenSette, 0.1 to 1.0 mL.....		each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet .....		50/pkg.....	21856-96
Pipet, volumetric, 10.00 mL, Class A.....		each.....	14515-38
Pipet Filler, safety bulb .....		each.....	14651-00
PourRite Ampule Breaker.....		each.....	24846-00

### *For Technical Assistance, Price and Ordering*

In the U.S.A. call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.